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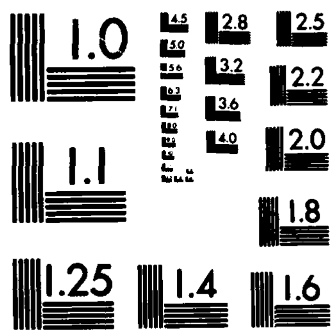
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SIZE DISTRIBUTION FOR G. P. ZONES IN DILUTE AL-ZN ALLOYS

BY

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EVANSTON, ILLINOIS

THE SIZE DISTRIBUTION FOR G. P. ZONES IN DILUTE AL-ZN ALLOYS

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Introduction

There is considerable current interest in the theory of particle coarsening. The original theories by Lifshitz and Slyozov (1) and Wagner (2) assume no particle interactions and therefore apply strictly only to the limiting case of zero volume fraction of second phase. Marqusee and Ross (3) have recently provided a very clear treatment of this problem. Sauthoff and Kahlweit (4) have taken into account variation of the diffusion coefficient with coherency, Ardell (5) introduced a modification which takes into account particle interactions, and the various possibilities for the diffusion geometry have been extended by Tsumuraya and Mayata (6). Particle encounters have been treated by Davies et al (7), and Brailsford and Wynblatt (8) have developed a theory that assumes that the growth of any particle is a function of the entire size distribution. The primary motivation for all these treatments is the fact that the experimentally determined particle size distributions do not agree with the LSW theory; in particular, they are broader than predicted, and do not exhibit the sharp cut-off at some maximum size. This topic is nicely reviewed in ref. 6, where much of the available data is considered (but see also refs. 9-11). The authors conclude that "the application of the models to experiment must await the aquisition of size distribution histograms in a variety of systems with reduced statistical errors." Glicksman and Voorhees (12) have just completed a new theoretical study via computer simulation, showing that the shape of the size distribution varies considerably with volume fraction of the second phase. At 0.5 or more, the assymetry to small sizes characteristic of the LSW theory and its various modifications vanishes, and is replaced by an asymmetry to large sizes. A similar result has been obtained by Margusee and Ross (13).

The Al-Zn system is particularly attractive for a comparison between theory and experiment because the G. P. zones that form during aging are coherent (with modest coherency strains), equiaxed, and randomly distributed. The volume fraction can be varied appreciably, and with small-angle scattering it is possible to obtain the entire size distribution from a large number of zones ($\sim 10^{16}$). Here we present such a study carried out with x-rays after various aging treatments, and after aging and fatigue to stimulate zone growth.

Experimental Procedures

The samples were made from alloy rods 3.5 and 5.3 at pct. Zn (which will form 2 and 4 volume percent zones (14)). Rolled and annealed strip, 0.8 mm thick, was given a strain of 1-2 pct. and lowered slowly into a salt bath at 803°K, to produce coarse grains 1-3 mm in size. A solution heat treatment at 698°K for 14 hrs. was followed by an air cool, reversion at 523°K, and reaging at various temperatures, from room temperature to 373°K. (A few samples were subjected to $\sim$ 10 cycles at 3.3 ksi before reversion, to reduce the tendency for grain boundary cracking (15).) Some specimens were also fatigued to stimulate zone growth. An Instron electrohydraulic machine was employed for this purpose, in pull-pull under strain control, or in push-pull (R=-1) with dynamic displacement control. Strains were measured with a clip-on extensometer, and both high- and low-cycle regimes were employed.

X-ray small-angle scattering was obtained with a Rigaku 12 kw generator operated in point focus with filtered MoK$_\alpha$ radiation, and a circular slit system (0.5 and 0.3 mm slits 250 mm apart to define the x-ray beam). In this way, the beam was considerably smaller than the grain size, and also no slit collimation corrections were required. The peak from a thin polystyrene sheet placed in the beam was employed to normalize different runs, and as well, to detect any changes

in the intensity of the incident x-ray beam during a set of experiments. Parasitic scattering was measured without the sample, and corrected for sample absorption prior to subtraction. Total ccunts accumulated ranged from 3000 to 10000 (taking 10 hrs. to 2 days per measurement), and the parasitic intensity was 15 pct. or less. A linear position sensitive proportional detector was used; its linearity and uniformity were carefully examined. Large tilts of a sample around the incident beam were made to see if the zones were truly spherical, since it is known that as the zones grow in this system, they can become ellipsoidal (16). No such case is included in the data presented here. Particle size distribution were obtained following Letcher and Schmidt (17) and Brill and Schmidt (18). As a check on the distributions, the moments that correspond to the Guinier and Porod radii were calculated, and compared to the values obtained directly from the data. Porod plots (for the most part) exhibited horizontal segments, indicating sharp interfaces at the zone boundaries. In many of the samples, the integrated intensity was the same before and after fatigue, indicating that coarsening was occurring. But some samples were studied in which the volume fraction increased during fatigue.

Fifteen different samples were examined and Table I summarizes the various treatments, integrated intensities, and average zone sizes. Further details can be found in ref. 19, and will be published in a paper concerned with the specific effects of fatigue on G. P. zones.

Results and Discussion

All of the size distributions superimpose, as shown in Fig. 1. This indicates that the measurements were made in the coarsening stage. [The superposition also implies that scaling holds.]

It is abundantly clear from Fig. 1 that despite the small volume fraction of zones, the size distribution is broader than that predicted by LSW theory, or any of its modifications. Also, the distribution is asymmetric toward large sizes, whereas theory predicts asymmetry to small sizes. None of the available theoretical treatments of coarsening can explain these results. The curve is also broader than a log-normal distribution. These alloys decompose inside the spinodal, and it appears that the diffusion paths are affected. Cahn (20) showed that a spinodally decomposed alloy would resist coarsening, and Weins and Cahn (21) found that if the particles surrounding any given particle were quite similar, the coarsening rate was reduced. While these treatments provide a qualitative explanation for our results, a more complete quantitative theory of coarsening is needed, to take into account the role of the initial distribution on the asymptotic solution. Topology and percolation may be important factors in controlling the diffusion paths. Many of the current theories assume the particles are points.

[The results in Table I also show that fatigue accelerates zone growth. We consider these results in more detail in the paper mentioned above.]

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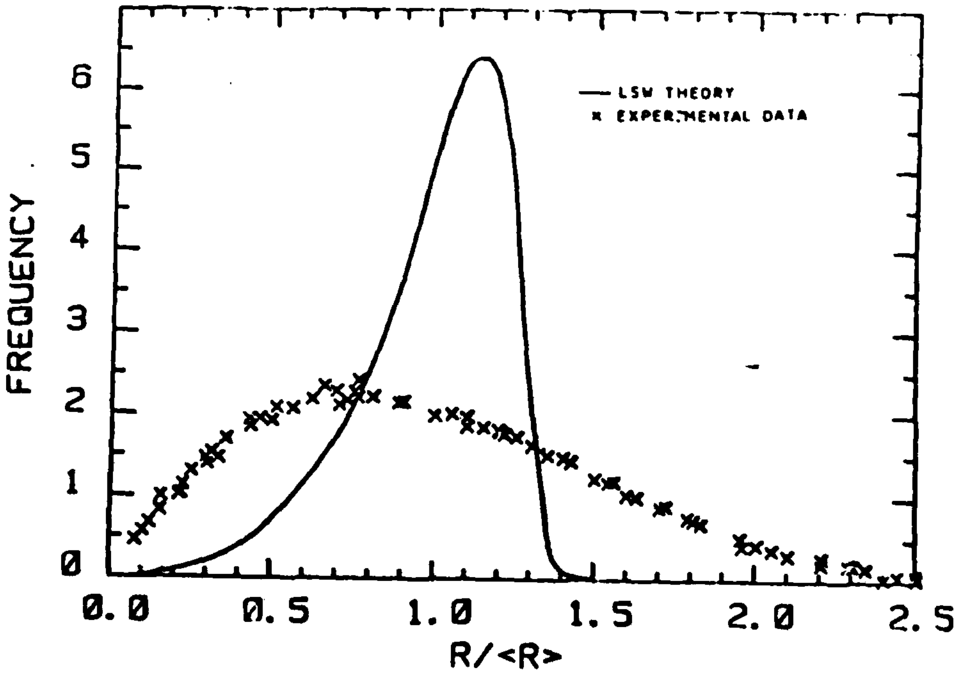

FIG. 1

Size distribution comparison, theoretical and experimental (x). (The x's include all 15 samples.)

TABLE I

Results of SAXS

SAMPLE (a/oZn)	HEAT TREATMENT[x]	FATIGUE $\sum_n \Delta\epsilon_{p\ell}$ (%)	R_{GLS}[o] (Å)	R_{GSD} (Å)	R_{PLS} (Å)	R_{PSD} (Å)	FLAT POROD REGION	⟨D⟩	Q(% RELATIVE TO FULLY AGED)
P(3.5a/oZn)	1	——	14.6	15.0	10.9	13.8	Yes	18.4	100
	2	.50	6.0	6.5	5.3	6.3		9.1	64
	2	7.0	9.0	8.6	6.3	8.3		11.2	100
	2	43.5	13.8	13.1	6.7	12.6		17.2	100
L(3.5a/oZn)	‡3	100 cycles	6.5	6.4	4.5	6.1		8.2	*
	‡‡3grainA	@ ± 14.5ksi	8.6	8.5	6.6	8.2		11.1	65
	3grainB	@ 77°K	9.8	10.0	6.0	9.7		13.2	100
S(5.3a/oZn)	1	——	19.3	20.3	15.4	17.5	Yes	22.5	100
	4	1.85	15.2	15.2	13.6	14.5		19.5	20
	4	8.60	17.6	18.0	13.7	16.7	Yes	22.7	61
	4	9.10	13.6	13.6	9.7	12.9	No	17.5	98
V(5.3a/oZn)	5	——	13.5	13.4	10.3	12.9	No	17.5	100
	5grainA	3.50	19.9	18.9	14.0	17.8	No	23.9	93
	5grainB	3.50	22.5	22.3	17.7	19.5	No	25.4	87
T(5.3a/oZn)	5	——	21.3	21.0	17.9	18.2	Yes	23.7	100

‡Aging occurs during 12 hr. SAXS measurement at 178°K.
‡‡Additional aging occurs during 1-2 days of further SAXS measurements at 298°K.
°LS is result of least square fit to Guinier or Porod approximation.
SD is result from the ratio of moments of the size distribution.
*The low temperature GP zone composition is not sufficiently well defined to estimate state of completion of aging.

x1. Homogenize at 698°K/14hr.; air cool; solutionize at 698°K/1 hr.; water quench to 298°K; age at 298°K/2 days.
2. Treatment #1 above plus: 2×10^4 cycles @ ± 3.3 ksi; reversion at 423°K/20mins.; re-age at 351°K/64hrs.
3. Treatment #1 above plus: reversion at 423°K/20mins.; re-age at 298°K/14 days.
4. Treatment #1 above plus: 2×10^4 cycles @ ± 3.3 ksi; reversion at 423°K/20mins.; re-age at 373°K/18hrs.
5. Homogenize at 698°K/14hrs.; air cool; reversion at 523°K/30mins.; re-age at 298°K ~ 6 months.

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13. ABSTRACT

The G.P. zone size distribution has been measured in dilute Aℓ-Zn alloys via small-angle x-ray scattering. This distribution does not agree with current theories for the Ostwald ripening process.

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